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RESEARCH OF OPTICAL DIELECTRIC MICROCAVITIES FOR DETECTING NANOPARTICLES

SUMMARY OF DISSERTATION

For the PhD HSE

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GENERAL CHARACTERISTICS

Problem statement

The issue of monitoring solid particles availability including nanoparticles in clean gas medium is critical in a large number of application areas from providing non-hazardous atmosphere for breathing of people including breathing during outer-space flights and to atmosphere control inside lithography machines manufacturing large-scale integration microcircuits with nano-sized elements. In the latter case, to exclude rejected material the issue is even about single particles.

Under the conditions of digital transformation of economic branch, plants of the future engineering and transition to Industrial Internet of Things there is a need to create not just high-sensitivity sensors but inexpensive, compact, needing no setting and maintenance ones. And if such a problem is solved for the large particles (>0,5 μm) on the ground of diffusion recording, then the solution for the nanosized particle range has not been found yet.

Existing devices for monitoring nanoparticles concentration are not sensitive to single nanoparticles, require difficult sample preparation, they are unhandy and expensive. A highly qualified operator is needed to work with such device.

Using the methods of nanoparticles recording based on the appliance of dielectric optical microcavities (DOM) allows creating sensors that meet the requirements of digital economy applicable to measuring systems.

Status and degree of development of the problem

At the moment a great number of research teams are studying the issue of nanoparticles detecting by the means of DOM. The team from the University of Wisconsin-Madison (USA) shows the possibility of detecting nanotubes of different sizes using facility assembled as a part of an electronic microscope. In its studies the team uses lithographic resonators with a Q-factor of 10^6 as well as the method of detecting a signal by frequency shift. The team from the Max Planck

Institute for the Science of Light, Germany, is one of the leaders in this field. In their research the team investigates biological objects in fluid. Great attention should be paid to their research on comparing sensibility indices of different detecting methods of objects of different sizes. Another interesting research team from Center for Photonics Technology, USA, made an attempt to detect nanoparticles in the air medium using cylindrical glass cavities. However the Q-factor is relatively low - 10^6 and therefore the sensitivity is far from great.

Purpose and objectives of research

The purpose of the study is to improve methods and monitoring facilities for nanoparticles concentration using dielectric optical microcavities.

To archive this purpose a number of objectives were set, among these are:

1. To study the methods of DOM manufacturing;
2. To study factors affecting the DOM metrological characteristics during manufacturing process;
3. To study the influence of DOM internal inhomogeneities on its metrological characteristics;
4. To study and develop optical communications methods which ensure the maximum Q-factor of DOM;
5. To study and analyse monitoring methods of nanoparticle number concentration in air medium;
6. To develop creation methods of nanoparticle number concentration for determining the metrological characteristics of the sensor based on DOM in air medium;
7. To study and develop methods for improving DOM sensibility in interaction with nanoparticles in a liquid medium.

Relevance of the study

At the present time DOM is widely used in science and technology. DOM is a high-quality selective element, which is an analog of a high-quality circuit or microwave resonator, having a narrow resonance curve the possibility of tuning in frequency and resistant to interference. They are used as frequency filters in which the tuning can be carried out due to mechanical compression or stretching and also due to a change in temperature. DOM allow to realize high-frequency signal generators with small phase noise. The literature describes the possibility of combining several microresonators in different configurations of fine line filters, the theoretical transmission width of the amplitude-frequency characteristic of such a filter is unlimited. Also multistage filters with a carrying capacity up to 100 GHz are manufactured on the basis of DOM.

In Jet Propulsion Laboratory, Pasadena, the USA uses DOM as part of an integrated gyroscope in satellites and spacecrafts. Sensors based on DOM are able to measure temperatures from cryogenic to room temperature with a resolution of 10-6 K. DOM can be used as add-on devices for stabilizing lasers. Through the connection with the DOM the laser emission spectrum narrows and it also becomes possible to create tunable in frequency lasers.

Resonators allow to create optical combs. The use of detectors of nanoparticles based on resonators is of particular interest because they are capable to increase significantly the sensitivity limit of modern methods for measuring the concentration of nanoparticles.

Nanoparticles of various materials are widely used in manufacturing, in science, and in medical research. Among them, the most common particles are Al2O3, TiO2, SiO2 and ZnO. Geometric dimensions, surface morphology, mass and counting concentration and charge are most often studied parameters of nanoparticles.

One of the important objectives in studying of nanoparticles is detecting ultra-small concentration of nanoparticles in different media. This objective can be achieved with the help of great number of procedures which can be divided into two groups: deterministic and probabilistic methods. Deterministic method includes scanning electron microscope, transmission electron microscopy, atomic force microscopy, piezobalance dust monitor. Probabilistic methods includes static light scattering, dynamic light scattering, differential mobility analyzer and ultrasound attenuation spectroscopy.

Using the above methods in most cases allows the measurement of one of the above nanoparticles parameters. Also on the market there are no devices that can work both in air and water media, with the exception of microscopy, where it is possible to do this through laborious preparation of the sample. Another significant disadvantage of all the indicated methods is the inability to measure ultra-small concentrations of nanoparticles up to 5 nm in size.

Another promising method is the use of optical diellectic microresonators with whispering gallery modes as detectors for nanoparticles. The microcavity is a body of revolution with diameters from hundreds of nanometers to tens of micrometers.

For the creation of DOM intended to nanoparticle detention two parameters are of particular importance: field confinement which determines the volume active region and Q-factor, which is directly proportional to the DOM sensitivity to the minimum concentration of nanoparticles.

Author’s personal contribution

The thesis contains the results of the work carried out by the author for 4 years. Personal contribution of the author to the thesis: ¶

1) setting of the purpose and objectives of the study;

2) analysis of literature and research methods;

3) designing, collecting and debugging experimental installations;

4) conducting experiments in full volume; ¶

5) processing the results and formulating the findings of the study.

The practical value of the work is that on the basis of this study a prototype DOM suitable for determining the concentration of nanoparticles in the air was created. Acts of implementation and 2 patents were received.

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Research methodology

We apply development methods of measuring instruments, empirical and statistical methods of studying metrological characteristics to theoretical research. Processing of measuring signals was carried out in the software packages MATLAB, WinPython, Origin.

We apply method of scanning electron microscopy, method of transmission electronic microscopy, method of differential electric mobility, method of dynamic light scattering, method of optical microscopy, method of confocal microscopy and method of interference microscopy to experimentation.

Thesis statements for defending

1. The formation of a homogeneous thermal field by circular heating in the presence of particles with a size of 0.5 μm and 1 μm and concentration to a maximum of 4×10^5 pc. and 8×10^4 pc. respectively ensures the production of DOM with a deviation of a diameter not worse than 10% in the range from 160 μm to 1800 μm, deviation of the plane of the equator tangent from the axial line of the leg is not more than 2% and a Q-factor of $1 \times 10^9 \pm 20\%$;

2. The presence of internal inhomogeneities of the refractive index *n* in DOM to a maximum of $\Delta n = 3 \times 10^{-3}$ and a diameter to a maximum of 10 μm does not affect its Q-factor;

3. The use of a fiber with a long parabolic overstretching provides maximum sensitivity of the nanoparticles sensor due to the high Q-factor of DOM not worse than $1\times10^9\pm20\%$;

4. The sensor based on DOM allows to detect TiO_2 particles with an integral number concentration to a maximum of $1,55*10^5\pm0,12*10^5$ U / cm^3;

5. To use DOM as a sensor of silver nanoparticles in a liquid medium, it is necessary to keep the resonators in distilled water for at least 60 minutes under normal conditions.

Scientific novelty

1. The procedure of manufacturing DOM from optical fiber and allowing to reproduce the geometrical dimensions of DOM with an accuracy of 10% with a Q-factor not less than $1\times10^9\pm20\%$ is developed;

2. For the first time in the world the index profile at the DOM center was obtained by means of optical tomography. It is shown that the presence of inhomogeneities of the refraction index $\Delta n=3\times 10^{-3}$ and a diameter of not more than 10 μm does not reduce the Q-factor less than $1\times10^9\pm20\%$ and does not affect its Q factor;

3. The procedure for fabricating a sublong-wavelenght optical fiber for communication with DOM having a special form of overstretching which allows to measure the Q factor up to 109;

4. The possibility of detecting small concentrations of TiO2 aerosol nanoparticles up to $(1,55\pm0,12)\times10^5$ unit/cm^3 in air medium using DOM has been experimentally demonstrated. It is shown that as a result of activation by high-temperature heating of siloxane bonds in polycrystalline quartz, adhesion of titanium dioxide nanoparticles to the DOM surface in the air medium occurs, which makes it possible to detect single nanoparticles of titanium dioxide;

5. The procedure for activation of siloxane bonds by interaction of the resonator surface with hydroxyl ions for at least 60 minutes is developed which ensures the adsorption of silver nanoparticles in a liquid medium.

General conclusions

1. It is shown that the circular heating of the optical fiber allows the creation of optical resonators with reproducible metrological characteristics. Practical significance is confirmed by the act of implementation;

2. It is shown that the internal inhomogeneities of DOM refractive index do not affect its metrological characteristics;

3. Developed procedure allows fabricating sublong-wavelength fiber with working length range from 3 to 15 mm provides a seamless transition between diameters, value of transmission coefficient is not less than 98.5%. The resulting fiber geometry possesses the necessary rigidity allowing to minimize the effect of the electric charge accumulated on the coupling elements of waveguide-resonator, thereby allowing a stable connection with the DOM. The scientific novelty is confirmed by the patent No. 52645040 and the registration of software No. 2016618965.

4. The manufacturing of sub long wave fiber with a parabolic form of stretching allows one to provide a connection with DOM, obtained by thermal and mechanical methods for the development of highly sensitive nanoparticle sensors.

5. Developed procedure of activation of DOM surface allows measuring the concentration of silver nanoparticles in fluid medium at a concentration of 0.05 ppm and higher;

6. It is shown that for the implying of sensors based on DOM for the detection of silver nanoparticles in water medium at a concentration of 0.05 ppm and above, hydroxylation of DOM in distilled at least 60 minutes water must be performed.

List of publications

1. Ivanov A.D., Min’kov K.N., Samoilenko A.A. Method of producing tapered optical fiber // Journal of Optical Technology. 2017, Vol. 84, Issue 7, pp. 500-503, Scopus, Q2.
2. Samoilenko A.A., Levin G.G., Lyaskovskii V.L., Min’kov K.N., Ivanov A.D., Bilenko I.A. Application of Whispering-Gallery-Mode Optical Microcavities for Detection of Silver Nanoparticles in an Aqueous Medium // Optics and Spectroscopy. 2017, Vol. 122, № 6, pp. 1002–1004, Scopus, Q3.
3. Ruzhitskaya D.D., Samoilenko A.A., Ivanov A.D., Min’kov K.N. Analysis of the Transmission Spectra of Optical Microcavities Using the Mode Broadening Method // Optoelectronics, Instrumentation and Data Processing. 2018, Vol.54, № 1, pp. 61-68, Scopus, Q4.
4. Min’kov K.N., Ivanov A.D., Samoilenko A.A., Ruzhitskaya D.D., Levin G.G., Efimov A.A. Measurement of Low Concentrations of Nanoparticles in Aerosols Using Optical Dielectric Microcavity: The Case of TiO_2 Nanoparticles // Nanotechnologies in Russia. 2018, Vol 13, № 1-2, pp. 38-44, Scopus, Q2.